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Determination of Apparent Viscosity of Bituminous Binders at Elevated Temperature by Rotational Viscometer

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Bitumen, Tar and Related Products Sectional Committee, PCD 06

FOREWORD

This Indian Standard was adopted by the Bureau of Indian Standards, after the draft finalized by the Bitumen, Tar and Related Products Sectional Committee had been approved by the Petroleum, Coal and Related Product Division Council.

Measurement of the non-Newtonian viscosity of a variety of bituminous material is important in various applications. Such measurements could be related to the determination of apparent viscosity of unmodified and modified binders at mixing and compaction temperatures, as well as at application temperature for bituminous emulsions and cutbacks. The non-newtonian viscosity can be measured at any given temperature by subjecting the sample to a shear rate sweep or steady-shear for a chosen torque level. This standard prefers viscosity measured using a chosen torque level.

Considerable assistance has been drawn from ASTM D4402-15 'Standard test method for viscosity determination of asphalt at elevated temperatures using a rotational viscometer' and EN 13302 : 2010 'Bitumen and bituminous binders: Determination of dynamic viscosity of bituminous binder using a rotating spindle apparatus' while formulating this standard.

The Composition of the Committee responsible for formulation of this standard is given at Annex A.

In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off , it shall be done in accordance with IS 2 : 1960 'Rules for rounding off numerical value (*revised*)'.

Indian Standard

DETERMINATION OF APPARENT VISCOSITY OF BITUMINOUS BINDERS AT ELEVATED TEMPERATURE BY ROTATIONAL VISCOMETER

1 SCOPE

This standard prescribes the method of test for measurement of apparent viscosity of modified and unmodified bituminous binders using a rotational viscometer and a temperature controlled thermal chamber for maintaining the test temperature.

NOTES:

1. In general, standard application temperatures are reported, although the dynamic viscosity can be measured at other temperatures, if required. Similarly, viscosity is generally reported at standard rates of shear, although additional measures can be taken at varying shear rates if required.

2. In this standard, the phrase "bituminous binders" represents unmodified and modified bitumen, bituminous emulsions and cutbacks.

2 TERMINOLOGY

For the purpose of this standard, the following definitions shall apply.

2.1 Newtonian Liquid — A liquid for which the rate of shear is proportional to the shearing stress. The constant ratio of the shearing stress to the rate of shear is the viscosity of the liquid. The viscosity of a newtonian liquid is therefore not dependent on its shear rate. If the ratio is not constant, the liquid is non-newtonian. Many liquids exhibit both newtonian and non-newtonian behaviour, depending on the shear rate or temperature, or both.

2.2 Shear Rate — The measure of the speed at which the intermediate layers of the liquid move with respect to each other. Its SI unit is sec^{-1}.

2.3 Shear Stress — The force per unit area required to produce the shearing action. Its SI unit is Pascal.

2.4 Viscosity — The ratio between the applied shear stress and shear rate is called the viscosity. For a newtonian liquid, this ratio is constant. For a non-newtonian liquid, this depends on shear rate and hence, called as apparent viscosity. The SI unit of viscosity is the Pascal second (Pa·s).

2.5 Form Factor — Specific factor or factors to be applied for the individual equipment in order to obtain the actual viscosity from the readings, mainly due to the geometry of the apparatus.

3 OUTLINE OF THE METHOD

The standard prescribes the method for measurement of apparent viscosity of bituminous binders using rotational viscometer. The torque on the apparatus-measuring geometry, rotating in a thermostatically controlled sample holder containing bituminous binders sample, is used to measure the relative resistance to rotation. The torque and speed are used to determine the viscosity of the bituminous binders.

NOTE — Comparisons between non-newtonian viscosity values of bituminous binders should be made only with similar conditions of temperature, shear rate, and shear history.

4 APPARATUS

4.1 Rotational Viscometer

Capable of measuring the torque required to rotate the selected apparatus measuring geometry at a selected constant speed while submerged in bituminous binders at constant desired test temperature, and with the capability to convert the torque measurement to viscosity.

NOTE — This calculation may need to be done manually for some instruments.

4.1.1 In order to cover all types of unmodified and modified bitumen, bituminous emulsions and cutbacks, under the most usual test conditions, the rotational viscometer should have sample containers and rotating spindles allowing following capabilities:

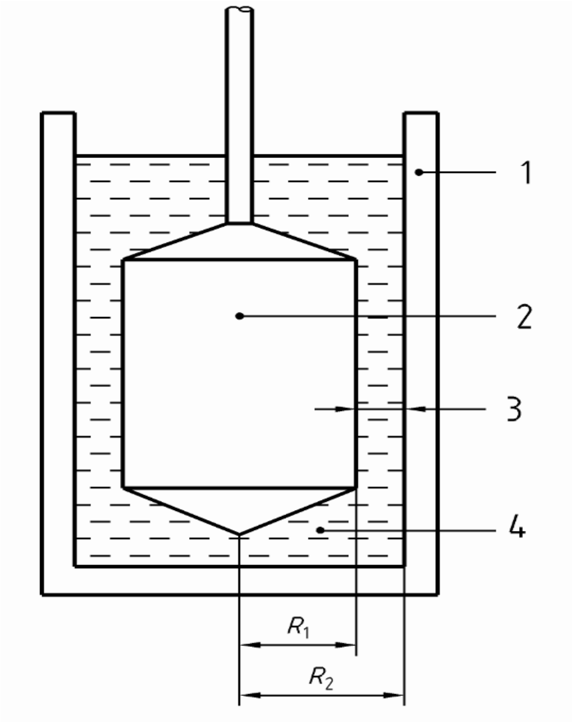
a) Range of shear rate : $1\ \text{s}^{-1}$ to $200\ \text{s}^{-1}$; and
b) Range of dynamic viscosities : 10^{-2} Pa·s to 10^{3} Pa·s

4.1.2 The following two conditions are indication of correct measuring geometry and may be used in general or in case of rotating cylinders:

a) Ratio of the radius : $R_2/R_1 \geq 1.1$ (*see* Fig. 1); and
b) Difference between the radius : $R_1 - R_2 = 1$ mm to 6 mm.

NOTE — The typical test temperatures are:

a) For bituminous emulsions — 40°C;
b) For cutbacks and fluxed bitumen — 60°C to 90°C; and
c) For unmodified or modified bituminous binders — 90°C to 180°C.



Key

1	Sample container
2	Spindle
3	Thickness of sample being measured
4	Sample under test
R_1	Inner radius (spindle)
R_2	Inner radius (sample container

FIG. 1 A TYPICAL ROTATIONAL VISCOMETER

4.1.3 The rotational viscometer shall be calibrated annually using a certified reference newtonian fluid of known viscosity at various temperatures to be used during the test. The viscosity measured shall be within ± 2 percent of the certified value, or else a calibration constant (viscosity of the calibration fluid/viscosity indicated by the apparatus) must be determined and applied.

4.2 Appropriate Spindle(s)

A spindle, or set of spindles, appropriate for the equipment, that will allow, for the materials to be tested and each specific test condition, dynamic viscosities to be measured with an accuracy at least equal to ± 5 percent.

NOTES:

1. If achievable accuracy is not known from the information given by the manufacturer of the equipment, the spindles should be selected so as to obtain dynamic viscosity readings greater or equal to 20 percent of the spindle working range for the chosen test conditions.

2. For low viscosity materials (typically, for viscosities below 100 mPa.s), it may be difficult to fulfill these requirements with existing equipment. Lower accuracy is then permitted but needs to be documented while indicating, in the test report, the actually achieved level of accuracy or, if not known, the position of the reading with regard to the working range of the used spindle.

4.3 Apparatus-Measuring Geometry

The part of the equipment that is immersed in the bituminous binder sample, the dimensions of which are used, in conjunction with the rotational resisting torque, to calculate the apparent viscosity.

NOTE — This geometry may be referred to the spindle, bob, inner concentric cylinder or vane.

4.4 Sample Container

Which allows the equipment to be used in coaxial mode and therefore enable the control of shear rate.

4.5 Temperature-Controlled Thermal Chamber

With least count 0.5°C and capable of maintaining the sample of bituminous binder at the test temperature between 90 and 210°C.

4.5.1 The accuracy of the temperature reading and the temperature stability of the temperature controller are to be checked at least every six months by placing a bituminous binder or high flash point oil in the test chamber, and equilibrating to a temperature within 50°C of the temperature(s) to be used during the test. The sample temperature shall then be measured to within ± 1°C. If any temperature differential is indicated, the set point of the temperature controller shall be offset accordingly.

4.6 Balance

With least count 0.1 g.

5 PROCEDURE

5.1 Preparation of Test Sample

The bulk binder sample shall be placed in an oven maintained at a temperature of about 80°C above the expected ring and ball softening point temperature or at a maximum of 200°C, whichever is the lower. Do not overfill or underfill the sample container, as the sample volume is critical to meet the system calibration conditions. The container shall be covered with a loose lid to protect it against oxidation. The test sample shall be fluid before transferring it to the sample container.

5.2 Preheating

5.2.1 Set the temperature control device to the desired test temperature.

5.2.2 Connect the selected spindle to the rotating spindle viscometer and lower it into an empty sample container placed into the temperature control device. Wait until equilibrium temperature is obtained (about 1 h). If, for practical reasons, the spindle cannot be preheated, it can be connected at ambient temperature and lowered into the hot sample. Temperature equilibration time then needs to be extended accordingly.

5.2.3 Additional sample containers may be preheated at the same temperature in a separate oven for 45 min.

5.2.4 Remove the sample container from the temperature control device or the oven and add the volume of sample specified for the spindle to be used. Do not overfill or underfill the sample container, as the sample volume is critical to meet the system calibration conditions. Avoid incorporation of air bubbles into the samples.

5.2.5 Place the loaded sample container into the temperature control device. Lower the spindle into the sample to the depth specified by the instrument manufacturer.

5.2.6 Ensure the equipment is level by use of a levelling device (for example, spirit level). Allow the system to equilibrate at the test temperature. Allow the moveable part to rotate during the pre-heating time at a very low shear rate. Maintain the temperature during the test at within ± 0.5°C up to 100°C and ± 1.0°C above 100°C.

5.2.7 Bring the bituminous binders to the desired temperature within 30 min and allow it to equilibrate at the desired test temperature for a minimum of 10 min before beginning the measurement.

5.3 Measurement

5.3.1 Start the motor rotation of the viscometer at a speed that will develop a resisting torque that is between 10 and 98 percent of the full-scale instrument capacity. The desired shear rate is achieved with a precision of ± 10 percent.

5.3.2 Maintain this speed and allow the sample to equilibrate for an additional 5 min. Temperature should not deviate more than ±1°C during this conditioning period. Measure either the viscosity or the torque at 1 min intervals for a total of three minutes.

5.3.3 Record the torque, viscosity (where necessary, apply the form factor to calculate dynamic viscosity from the reading of the instrument), shear rate, and temperature.

5.3.4 Repeat steps **5.3.1** to **5.3.3** for each test temperature required.

NOTES:

1. Reuse of sample is allowed for successive measurements at different temperature levels, as long as the total time for the hot sample does not exceed 4 h.

2. If torque readings are above 98 percent of the instrument capacity at the lowest test temperature, decrease the speed of rotation of the apparatus-measuring geometry and continue with the test, or repeat with a smaller diameter geometry and the appropriate volume of sample.

3. If the torque reading is below 10 percent of the instrument capacity at the highest test temperature, increase the speed of rotation of the apparatus geometry, or repeat with a larger diameter geometry and the appropriate volume of sample.

6 CALCULATION

Calculate the arithmetic average of the three readings taken at 1 min intervals, rounded to three significant figures.

NOTE — If the instrument automatically averages the three readings, manual calculation is not needed.

7 REPORT

7.1 Report viscosity value with test temperature. Express the dynamic viscosity, in Pascal-second or milli-Pascal-second as the mean of two results, obtained on two separate samples. If the individual results differ by less than 10 percent, report the mean value of the two results. If the individual results differ by more than 10 percent of the mean value of the two results, the individual results and the mean value shall be reported.

7.2 Report apparatus measuring geometry type and size, torque in milli-Nm or percent of instrument capacity, and speed in s^{-1} or r/min.

8 PRECISION

The following criteria shall be used for judging the acceptability of any result (95 percent confidence level):

8.1 Repeatability

Duplicate values by the same operator using the same test equipment, in the shortest practical period of time shall be considered not equivalent if the difference in the two results, expressed as a percent of their mean, exceeds 3.5 percent.

8.2 Reproducibility

The values reported by each of two laboratories, representing the arithmetic average of duplicate determinations, shall be considered not equivalent if they differ by more than 14.5 percent.

ANNEX A

(*Foreword*)

COMMITTEE COMPOSITION

Bitumen, Tar and Related Products Sectional Committee, PCD 06

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